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PRINCIPLES OF HIGH TEMPERATURE MICROSCOPY

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PRINCIPLES OF HIGH TEMPERATURE MICROSCOPY

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An extensive literature survey is presented which deals with (1) the design of microscope objectives suitable for high temperature work, (2) the design of high temperature microscope stages and furnaces, and (3) the problems of high temperature photomicrography. Microscope systems are available having long working distances and with numerical apertures greater than 0.4. These systems have proven suitable for studying microstructure at temperatures above 750 C. Numerical apertures as low as 0.1 are suitable for observation of macrostructures and specimens in image-type furnaces. Several types of excellent furnaces are commercially available. Experimental data are given on the use of cameras with the American Optical Company high temperature microscope.

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PRINCIPLES OF HIGH TEMPERATURE MICROSCOPY

INTRODUCTION

Advances in high temperature technology await quantitative information concerning the behavior of matter at elevated temperatures. This vital data can be most easily and accurately obtained by direct observation or photography of the high temperature phenomenon.

The purpose of this paper is to survey the field of high temperature microscopy and add to previous reviews the experience of more recent work. Since the temperature range from room temperature to 750 C has been adequately treated by Chamot and Mason⁽¹⁾ and McCrone⁽²⁾, it will not be considered. Instead, this paper will treat systems capable of studying phenomenon above 750 C to the limit of existence of elemental solids.

GENERAL HISTORICAL REVIEW

The application of microscopy to high temperature research dates back to 1865, when Schulze (according to Chamot)⁽¹⁾ constructed a heating stage consisting of a bar of copper heated by a Bunsen burner flame. Significant work was done by Oberhoffer in 1909⁽³⁾, Robin in 1912⁽⁴⁾, Rogers in 1931⁽⁵⁾ and Esser and Cornelius in 1933.⁽⁶⁾ These investigators employed conventional microscopes and were thus restricted to moderate temperatures. The reader is referred to the treatise by Campbell⁽⁷⁾ and the papers by Baumann^(8, 9) and Reinacher⁽¹⁰⁾ for a general survey of the field.

OBJECTIVE DESIGN

Limitations on the Numerical Aperture

The diameter of the smallest specimen detail that can be resolved using a given objective is given by

$$Z = \frac{\lambda}{k (N. A.)} \quad (1)$$

In this equation N. A. is the numerical aperture, λ is the effective wave

length of the light forming the image and k is a constant approximately equal to two. Thus large values of N. A. indicate objectives which will resolve the finest detail. The N. A. is also related to the diameter of the objective and its working distance. Let n be the index of refraction of the object space (usually 1) and θ be one half of the angle included in the cone of light entering the objective from an object point. The numerical aperture is

$$\text{N. A.} = n \sin \theta. \quad (2)$$

In Figure 1, if ρ is the radius of a cross section of the cone of light at the objective and p is the working distance, then

$$\frac{\rho}{p} = \tan \theta. \quad (3)$$

Thus large N. A. values mean that the corresponding diameter of the objective must be large or the working distance shorter. In practice the largest value of the numerical aperture obtainable is about 1.6.

The maximum useful magnification of a well designed objective when used for visual observations may be taken to be 1000 times the N. A. ⁽¹¹⁾

Most furnace designs permit the use of windows which do not limit the N. A. of available long working distance objectives. Solar and arc image furnaces are the exception. In Figure 2, the sample must be viewed through a hole in the center of the concentrating reflector. A microscope of large numerical aperture would shade the sample from a correspondingly large cone of converging radiation and consequently decrease the maximum temperatures attainable in the furnace. The large heat fluxes involved also dictate that the working distance be great. Typical objective designs applicable to image type furnaces have working distances of about five inches and numerical apertures less than 0.2.

Required Depth of Field

An objective of high numerical aperture used to observe a rough surface may be incapable of focusing on all levels of the object. The depth

of field (d) is related to the numerical aperture as follows:

$$d = \frac{\lambda \sqrt{n^2 - (\text{N. A.})^2}}{(\text{N. A.})^2}.$$

Thus, if high resolution is required, the N. A. must be large and the object must have a polished surface. If the object is rough and all levels must be viewed, the depth of field must be increased by decreasing the N. A.. Objectives of low N. A. are generally less expensive than those of high N. A.. Therefore the choice of objective will depend on the intended application and may dictate the selection of an objective of relatively low N. A..

Objective Lens Systems

When studying the behavior of materials at elevated temperatures, it is necessary to consider how the microscope objective can be protected from excessive heating. This can be accomplished in three ways:

1. Use small samples. The heat content of these samples is small compared to that required to damage the objective. Conventional 16 mm objectives have been used successfully to observe small samples at temperatures up to 1750 C. ⁽¹²⁾
2. If large samples must be used, design the furnace so that parts near the objective are cooled by either flowing water or a gas stream. Cooling of the objective lens might be tried but is the least practical of the two alternatives.
3. Separate the sample and objective as much as is practical. This requires the design of unconventional, long working distance objectives. These unconventional microscope objective systems may be classified into five groups and will be considered in order: ⁽¹³⁾
 - a. Modified conventional objectives
 - b. Reflecting objectives
 - c. Reflecting objectives with zero or low power lenses
 - d. Catadioptric systems
 - e. Unit or low magnification systems

Modified conventional objectives

There are two commercially available microscope objectives of this type. One is a part of the Leitz heating microscope sold in this country by E. Leitz, Inc., 468 Fourth Avenue, New York 16, N. Y. This microscope objective gives only 1.5 X initial magnification. The second commercially available objective is the Unitron FF40X (Figure 3). It is sold by the Instrument Division of United Scientific Company, 204-206 Milk Street, Boston 9, Massachusetts. The FF40X has a working distance of 5.8 mm and a numerical aperture of 0.62. It is designed especially for use with the Unitron vacuum heating stage. Several fine photographs of 0.6 per cent carbon steel at 200 X magnification, with temperatures up to 1100 C, are shown in a brochure available from this company.

Reflecting objectives

This type of objective was used as early as the time of Newton. Early objective designs required a second mirror which obstructed much of an already small numerical aperture. Reflecting objectives were not used effectively until 1943, when Burch⁽¹⁴⁻¹⁶⁾ applied the Schwarzschild analysis to microscope systems. One objective of his design and construction has a N. A. of 0.65 with a working distance of 13 mm. Burch's design calls for aspheric surfaces, but he has shown that for 0.5 N. A. or less two spherical mirrors can perform satisfactorily. Figure 4 shows the design data for the Burch bi-spherical, monocentric mirror system. These systems have been used by Dewhirst and Olney⁽¹³⁾, Drew⁽¹⁷⁾, and Steenstrup.⁽¹⁸⁾ Dewhirst and Olney also refer to the use of this type of objective by Hershgorin⁽¹⁹⁾, Brumberg and Shevchenko⁽²⁰⁾, Brumberg⁽²¹⁾, Bennett, Woernley and Kavanagh⁽²²⁾, and Seeds and Wilkins.⁽²³⁾

It is possible to scale up the Burch design to obtain almost any desired working distance. Designs for objectives of 0.4 N. A. with working distances as great as four inches have been proposed.⁽²⁴⁾ It should be noted, however, that the objective becomes rather large and correspondingly expensive.

The objective used by Steenstrup was made by Bausch and Lomb Optical Company. Provisions were made in the complete microscope system (Figure 5) for simultaneous observation and photography. Vertical illumination, using a pellical mirror mounted behind the objective, provides sufficient intensity to bring out specimen details at 2400 F. A 1000-watt mercury arc lamp and condenser system direct the light on the pellical mirror. The mirror transmits image-forming light from the objective to the eyepiece. The objective (0.4 N. A. with 18 mm working distance) is designed for an initial magnification of 20 X. A similar conventional objective would have a working distance of only 3 mm.

Reflecting objectives with zero or low power lenses

The Jenkins, Buchele and Long⁽²⁵⁾ reflecting objective (Figure 6) has 0.5 N. A. with 11 mm working distance and has been used in a metallograph. A plano-convex lens was added to the objective to compensate for the longitudinal chromatic aberration introduced by the furnace window. They used a primary magnification of 100 X together with a 5-power eyepiece. Kohler-type vertical illumination was provided by imaging a zirconium arc light source on the convex objective mirror. Exposure measurement and control, simultaneous viewing, and 35 mm motion picture photography were provided. The Jenkins, et al. report includes photographs at 330 X of a 0.35 per cent carbon steel at temperatures up to 1650 F.

Catadioptric systems

Considerable work has been done by Grey⁽²⁶⁻²⁹⁾ on catadioptric Newtonian and Schwarzschild systems of low obscuring ratio for the purpose of obtaining an objective achromatic from 220 m μ to the near infra-red. These systems do not appear satisfactory for high temperature work because of the relatively short working distances.

Unit or low magnification systems

Dyson⁽³⁰⁻³³⁾ and Heal⁽³⁴⁾ have developed unit-power, reflecting relay systems which form a real image for examination with a conventional microscope. Dyson's design uses a spherical primary mirror and a partially reflecting flat. The unsilvered surface of the flat is slightly convex. This system permits a working distance of 11 mm with a N. A. of 0.57.

A relay system (Figure 7) is sold in this country by Edmund Scientific Corporation, Barrington, New Jersey (Catalogue 579, p. 36, Stock No. 50,038). A working distance of 12.8 mm and an N. A. of 0.574 are provided. It may be fitted with either a 4 mm or 8 mm conventional objective.

A system designed by Baumann^(8, 0) (Figure 8) for the study of phenomena above 1500 C forms an image in the object plane of a conventional microscope using a specially designed lens. The American Optical Company has improved this design and now offers a system of 0.1 N. A. and approximately 6-3/4 inches working distance. A power changer permits a rapid selection of one of three possible magnifications for each of three possible eyepieces. The system provides for mounting cameras and permits viewing of the specimen while taking pictures. An adjustable diaphragm located near the relay lens controls the amount of light reaching the film. Provision is also made for the use of filters.

FURNACE DESIGN

Microfurnaces or hot stages capable of use with the optical systems and microscopes discussed above are cited in the literature. They can be classified according to the method of specimen heating employed, namely:

1. Self-resistance heating
2. Metal or ceramic heating element
3. Electron bombardment heating
4. Radiation heating
5. Combustion heating

Self-Resistance Heating

Steenstrup⁽¹⁸⁾, Chalmers, et al.⁽³⁵⁾, Williams⁽³⁶⁾, and Cech⁽³⁷⁾ describe hot stages in which the specimen is self-resistance heated. The Steenstrup furnace (Figure 5) permits the heating of specimens in a vacuum or in a gaseous atmosphere. The unit is designed so that a specimen can be pulled in tension or forced into compression while at temperature. A nozzle directs a gas stream across the observation window to prevent clouding. Temperatures are read with a chromel-alumel thermocouple welded directly to the specimen.

The Chalmers furnace is most conveniently used with a Vickers projection microscope. The specimen can be heated in a vacuum or gas atmosphere. A thermocouple is used to measure the specimen temperature. The maximum reported operating temperature is 1000 C. In the William's furnace (Figure 9), the specimen can also be heated in a vacuum or in a gaseous atmosphere. A chromel-alumel thermocouple is used in conjunction with a radiation-type pyrometer for measuring the specimen temperature. The furnace can be used with any standard metallurgical microscope equipped with a vertical illuminator and a photographic attachment. The Cech microscope stage permits the observation of metals from the temperature of liquid nitrogen to an upper temperature determined by the specimen's melting point. The stage consists of a vacuum-tight, water or liquid nitrogen-cooled, copper chamber in which a specimen is heated by passage of an electric current. Temperatures are determined by means of a chromel-alumel thermocouple welded to the specimen. Above 1200 C a platinum alloy couple is used. The stage can be used with any conventional bench microscope.

Metal or Ceramic Heating Element

Various platinum, platinum alloy wound, or platinum strip furnaces have been described by Jenkins, et al.⁽²⁵⁾, Bridge⁽³⁸⁾, Endell⁽³⁹⁾, Mott and Ford⁽⁴⁰⁾, Pfeiffer⁽⁴¹⁾, Milne⁽⁴²⁾, and Speich, et al.⁽⁴³⁾ The platinum

wire is usually No. 20 AWG (0.032 inch in diameter) and is wound on alumina or alundum cores. Chromel-alumel or platinum vs. platinum-10 per cent rhodium thermocouples are used for temperature measurement. All the above furnaces can heat specimens in a vacuum or controlled gaseous atmospheres. Maximum temperature is limited to 1500 C.

Welch⁽¹²⁾ and Ordway⁽⁴⁴⁾ have described unique hot stage micro-furnaces in which the hot junctions of the platinum thermocouples are used as the furnace heating element. The heating current is isolated from that of the thermocouple by either a filter, in case of high frequency currents, or a vibrating switch for 50-cycle current. Temperatures up to 1600 C are claimed for this instrument. It is particularly suitable for observing the growth of small single crystals for X-ray diffraction measurements.

Esser and Cornelius⁽⁶⁾ describe a vacuum heating stage for use with a conventional Leitz metallograph. The resistance heating element is a nichrome wire wound on a ceramic core. A platinum alloy thermocouple is used to measure the temperature of the specimen. A furnace similar to the one constructed by Esser and Cornelius is described by Marechal and Douchet.⁽⁴⁵⁾ A nichrome strip furnace for operation up to 1000 C using a standard bench microscope is described by Roberts and Stadnichenko.⁽⁴⁶⁾

The Kofler hot stage⁽⁴⁷⁾ for use with a conventional microscope is available for the observation of specimens heated to 750 C. Kofler also makes a "Universal" hot stage which will heat specimens to 1500 C.⁽⁴⁸⁾ The Leitz Company⁽⁴⁹⁾ manufactures a hot stage which will heat specimens to 1800 C for use with their conventional microscope and metallograph. The application of this type of instrument to studies of the deformation and fusion characteristics of Seger pyrometric cones has been described by Mann.⁽⁵⁰⁾

The Unitron Company⁽⁵¹⁾ manufactures a vacuum or protective atmosphere heating stage. The chamber is made of stainless steel with an upper and lower portion bolted together. A rubber gasket maintains a tight seal between the two portions. Circulating water in both parts of the stage protects the gaskets and stage from overheating. The heating element is a tungsten coil. The specimen is observed through a quartz window in the bottom of the stage. Facing the specimen is a second quartz window which can be moved by an external lever, allowing a clear viewing surface at all times. The maximum temperature (1100 C) is obtained with a power of 400 watts and is limited by the materials of construction. A built-in platinum vs. platinum-13 per cent rhodium thermocouple is used to measure the temperature of the specimen. The stage is designed for inverted objective microscopes.

Molybdenum-wound furnaces have been described by Rogers⁽⁵⁾, Nichols⁽⁵²⁾, Saller, et al.⁽⁵³⁾, and Reinacher.⁽⁵⁴⁾ A tungsten-wound furnace has been described by Dewhirst and Olney.⁽¹³⁾ Newkirk and Bates⁽⁵⁵⁾ described the tungsten filament furnace of Figure 10. The above furnaces are all water cooled and specimens are heated under reduced pressures or in a controlled, gaseous atmosphere.

The American Optical Company will supply globar and graphite resistance-type furnaces for use with their Model II microscope.⁽⁵⁶⁾ The Globar, silicon carbide, heating element furnace is standardized on two matched bars operating in parallel. These bars are 12 in. x 5 in. (the heating length) x 1/2 in. With an input of about 8 KW and approximately 32 volts, temperatures up to 1500 C are obtained. In the carbon-type resistance furnace, the resistor is a 3/8-inch diameter electrode cut to 2-1/4-inch length with a 1/8-inch hole in the center for a reaction chamber. Approximately 7 KW at 17 volts are required to heat such a resistor to 2500 C. The carbon resistor is held between larger carbon blocks which in turn have water-cooled terminals. This furnace is

enclosed for atmosphere control and the entire jacket is water cooled. Recently, Rys, et al. ⁽⁵⁷⁾, published an extensive article describing a low-high temperature microscope stage which is presumably commercially available.

Electron Bombardment Heating

Ogilvie and Brinson ⁽⁵⁸⁾ describe a unique vacuum furnace in which the specimen is heated by electron bombardment from a tungsten filament. The specimen is supported on three pointed wires, two of which are the elements of a thermocouple. A chromel-alumel thermocouple is used to measure the temperature of the specimen. The furnace can heat to 1000 C at very low power levels.

Radiation Heating

Null and Lozier ⁽⁵⁹⁾ describe an arc image furnace which employs two elliptical mirrors. The furnace is capable of 3000 watts of radiant flux at an image within a circular area of 30 mm diameter. The center of image irradiance is as high as 15 watts per mm², and the flux density is equivalent to the radiation from a black body at more than 4000 K. Atmosphere control is achieved by means of a 2-1/2-inch diameter, transparent, Vycor or fused quartz cylinder. The cylinder, placed coaxially with the optical axis, surrounds the image focal position and extends through the central hole in the elliptical reflector. An American Optical Company thermal microscope was used with arc image furnace.

Newkirk and Brenden have reported an arc image furnace which employs two parabolic mirrors. ⁽⁶⁰⁾ While the unit does not develop the heat flux or temperatures that are attained in the Null and Lozier furnace, it is very suitable for studying the behavior of ceramic fuel materials at temperatures up to the melting point of uranium dioxide. The microscope used with this furnace is also an American Optical Company instrument.

Combustion Heating

Lambeth⁽⁶¹⁾ has described a hot stage with a blowpipe that burns a mixture of coal gas and air (or oxygen), hydrogen and air (or oxygen), or other suitable fuel. The furnace may be used with a conventional bench microscope and will attain temperatures up to 1600 C, depending on the fuel mixture.

PHOTOMICROGRAPHY

Photomicrography at room temperature has been extensively treated in many textbooks⁽⁶²⁻⁶⁷⁾ and articles⁽⁶⁸⁻⁷³⁾. However, photographing objects at high temperatures through a microscope has been discussed only briefly.^(8, 9, 74-77) Although the basic principles and practices of high temperature photomicrography are essentially the same as at room temperature, some special techniques are necessary.

Mounting

Any vibration between the furnace, microscope and camera is greatly amplified. Hence, it is necessary to minimize or eliminate all ordinary vibrations. A base of several tons of concrete would approach the theoretical ideal in eliminating vibrations. However, this is generally not practical and small vibration-damping devices must be employed. A single sturdy base, set on shock-absorbing supports (such as Lord, Thomson, or Vibrashock (Robinson)-type mounts), is effective for most work.

Cameras

The selection of a camera will depend upon the amount and variety of photomicrographic work. Almost any camera can be adapted to the microscope. The most common arrangement uses both the camera lens and the microscope eyepiece. The camera is focused on infinity and the microscope adjusted until the image is focused on the film plane. This arrangement is least sensitive to microscope-to-camera vibrations. The film magnification varies as the ratio of the focal length of the camera lens to that of the eyepiece.

Thus, if 50-mm-focal length camera lens is used with a 10 X eyepiece (25-mm focal length), the image on the film is twice the size of the microscope image at the field stop of the eyepiece.

Alternately, the microscope eyepiece may be used to project an image on the film plane with the camera lens removed. The magnification then depends upon the ratio of the projection distance to the focal length of the eyepiece.

Roll film and 35-mm cameras are readily adapted for use with the high temperature microscope. These cameras can be loaded and unloaded in the light and film processed by a photofinisher. No darkroom facilities are necessary. Single-lens reflex cameras are convenient for obtaining a good focus through the microscope.

Motion pictures are almost as readily obtained through a microscope as still photographs. Many reactions occur rapidly at high temperatures; others occur very slowly and a time lapse record is required. A motion picture record, observed repeatedly at varying projection rates, may reveal motion that direct visual examination omits.

The ideal motion picture camera would: (1) be driven by a variable-speed electric motor or a spring strong enough to expose a useful length of film, (2) be equipped with a single-frame release to be operated with a time-lapse attachment, (3) have interchangeable magazines for rapid loading, and (4) have a variable shutter for special exposures and a range of camera speeds.

The superior results obtained more than compensate for the extra cost of a camera with these features. Additional equipment could include a detachable lens system with a beam splitter or observation eyepiece. Some microscopes (e. g. , the A. O. high temperature microscope) are equipped with their own observation eyepiece. In addition, simultaneous data recording units are available from Par Products Corporation to record time, temperature, etc. on a portion of the film.

The technique of high speed photography may also be applied to the high temperature microscope for events too rapid for the eye to perceive. Time magnifications up to 1000 times (16,000 fps) can be provided with Fairchild, Wollensak, or Kodak high-speed cameras.

Time lapse motion picture records are especially useful in high temperature microscopy. Actions which take hours, days, or even weeks to occur can be accelerated on the screen. A camera equipped with a single-frame release is required. Controllers to actuate this release are commercially available from a number of companies, some of which are listed below.

Sample Engineering Co.	17 N. Jefferson, Dannville, Ill.
Par Products Corporation	926 N. Citrus Avenue, Hollywood 38, California
National Cine Equipment Co.	209 W. 48 Street, New York 36, New York
Opplem Company	83 Uhland Street, East Rutherford, New Jersey
Rolab Photo Science Laboratories	Sandy Hook, Connecticut
Stevens Engineering Co.	2421 Meblary Avenue, Los Angeles 64, California

Intervals from 1 to 1200 seconds per frame are available. In general, the controller consists of a solenoid which is actuated by an electric timer. When operating properly, the equipment can be left unattended.

Exposure

The most difficult part of high temperature photomicrography is proper exposure. At room temperatures (up to 600 C) a light source is required to illuminate the subject. However, when the specimen and often the heating element are emitting light, proper film exposure is complicated. Each film type is color balanced for exposure with a specific light source. At room temperature, the illuminator is the primary concern. Filters are

used to balance the known color temperature of the illuminator and that required by the film. The sample and heating element are an additional source of light at high temperatures. This light generally has a color temperature different from the reflected light from the illuminator.

The simplest and most direct method to determine the desired compensation from an incandescent light source and/or self-illumination is to make a series of exposures with unfiltered light. From this test exposure one can make corrections. For color film, a bluish filter is required if the film image appears too red and a yellowish filter if it appears too blue.

Once the proper exposure has been determined, it is necessary to duplicate it for later exposures. This is best accomplished with the microphotometers and exposuremeters which can be adapted to any standard microscope. They are generally self-contained with their own microammeter. A light-sensitive photocell is placed in the light path of the microscope between the eyepiece and the camera. Readings are arbitrary and must be evaluated for the microscope, magnification, and film. This is easily done through a series of test exposures for each film. It is possible to estimate the exposure of one film from another by comparing their ASA numbers. However, because of the different color sensitivity of films, this is only an estimate and will have to be adjusted as indicated by further experience.

If the temperatures and light values are changing during a photographic sequence, it may be necessary to change the exposure or add filters. This requires a light reading between or during exposures.

Photography with Still Cameras and Time-Lapse Motion Pictures

It is necessary to have a beam splitter or observation eyepiece incorporated into the equipment for motion picture work. Such a device is a convenience even in still photography. Using a small percentage of

the light that would enter the camera, test exposures can be made with the photocell from the light values through the beam splitter. This provides a continuous reading of light values. These values can be used to change the film exposure as the light from the object is changing. The beam splitter may also be used to view the object while it is being photographed.

The auxiliary lighting of a specimen becomes more difficult as the temperature of the specimen is increased. At temperatures from 650 to 1500 C, the illumination of a surface is the same as that for room temperatures. However, as the temperature increases, the intensity of the illumination also must increase. Although the amount of light available through the microscope is sufficient, the contrast over the object will decrease. To obtain sufficient detail at high temperatures, it is necessary to use a carbon or zirconia arc light source. The greater the light intensity, the higher the resolution of the surface. Above 1500 - 1600 C auxiliary lighting is of no value. Only differences in the body's temperature and the emissivity of the surface allow an observation of the surface details. It is necessary to view the object against a low temperature background. This insures sufficient contrast of the object's outline.

Films

The photographer has a wide choice of films which are suitable for photomicrography both in color and black-and-white. They can be obtained in a variety of sizes, emulsion speeds, grainsize and color sensitivity. The choice will generally depend on the results desired.

In general, it has been found convenient to use Plus X reversal black-and-white or Kodachrome A color film.

High Temperature Photography

Our experience has been primarily with the American Optical Company microscope which has been used with a carbon-arc image furnace and a

tungsten filament furnace (Figure 11). The microscope mounting consists of a table, bench, and a carriage. The table, 84 inches long by 36 inches wide, supports the bench and carriage. The bench is fabricated from two-inch thick steel bar stock and is 72 by 24 inches. It is supported on each corner by four tennis balls arranged in a tetrahedron to dampen the vibration. This mounting, originally suggested by Baumann⁽⁸⁾, has been very satisfactory. The bench can be lifted from the tennis balls by four manually-operated screw and hand wheel assemblies located near each group of tennis balls.

The carriage consists of a base and a platform which is pivoted on one end. The carriage, driven by a rack and pinion, slide along the length of the bench on flat-and "V"-type ways. The microscope, attached to the platform, can be adjusted in a plane perpendicular to the base by a hand wheel and screw. When the vertical and horizontal movements incorporated in the microscope are coupled, motion is possible along three co-ordinate axes. A photograph of a specimen of UO_2 being heated in the tungsten filament furnace is shown in Figure 12.

Magnification and field image-size data for two cameras which are currently in use are shown in Table I. The magnification data were obtained by photographing a stage micrometer and measuring the spacings on the film with a traveling microscope.

Exposures of Plus X, Kodachrome Type A and Daylight 16-mm motion picture film, Plus X and Ektachrome Type F 35-mm film have been calibrated for the two furnaces and are summarized in Table II. These data were obtained by sighting on the specimen through the extension tube of the microscope to which the camera is mounted. The brightness of the specimen is measured with an SEI exposure photometer.⁽⁷⁸⁾ A CdS-10 photocell⁽⁷⁹⁾ was mounted on the eyepiece of the microscope and its resistance measured with a volt-ohm meter. The resistance of the CdS cell was correlated with the apparent brightness of the specimen and subsequently

used to indicate the required filters to obtain proper exposure. When filtering was necessary, neutral density filters were generally used. In all cases, the camera lens and the microscope eyepiece were used in the exposure calibration. The microscope erect-image tube was removed when the 35-mm camera was used.

CONCLUSIONS

A number of different furnaces and microscope systems have been developed independently by various investigators. These systems permit effective observation of high temperature phenomena. Several excellent furnaces and microscopes have become commercially available. Microscope systems of long working distance and of 0.4 N.A. or greater are suitable for studying microstructure at temperatures above 750 C. Numerical apertures as low as 0.1 are suitable for the observation of specimens in image-type furnaces and for the observation of macrostructures (dimensions of 0.001 inches and larger).

A photograph provides a valuable record of high temperature reactions. The value of such a record justifies the use of motion picture cameras for high speed, normal, or time-lapse photography. Accurate exposure control requires the use of photometers to continuously monitor the light level. The most suitable microscopes provide for simultaneous observation and photography.

FUTURE WORK

Future work in high temperature microscopy will be concerned with applications of existing equipment to problems in high temperature technology. Emphasis might also be concentrated on new and novel receiving and recording devices. Composite pictures could show the energy radiated by the specimen in widely separated bands extending from infra-red through the ultra-violet. This would provide "color" pictures which possibility would show details of reactions at high temperatures not detected by present systems.

TABLE I
MAGNIFICATION CALIBRATION
FOR AMERICAN OPTICAL COMPANY MICROSCOPE

Visual Observation			Kodak Cine Special II				Exacta Camera	
Eyepiece	Power Changer	Magnification	25 mm Lens		50 mm Lens		58 mm Lens	
			Mag.	Field (mm)	Mag.	Field (mm)	Mag.	Field (mm)
7 X	2.5	17	1.75	4.8	3.5	2.4	4	2
7 X	5.	35	3.5	2.4	7	1.2	8	1
7 X	10.	70	7.	1.2	14	0.6	16	0.5
10 X	2.5	25	2.5	3	5	1.5	6	1.5
10 X	5.	50	5.	1.5	10	0.8	12	0.8
10 X	10.	100	10.	0.8	20	0.4	24	0.4
20 X	2.5	50	5.	1.5	10	0.8	12	0.8
20 X	5.	100	10.	0.8	20	0.4	24	0.4
20 X	10.	200	20.	0.4	40	0.2	48	0.2

Maximum resolution at 100 X - 0.0001 inches

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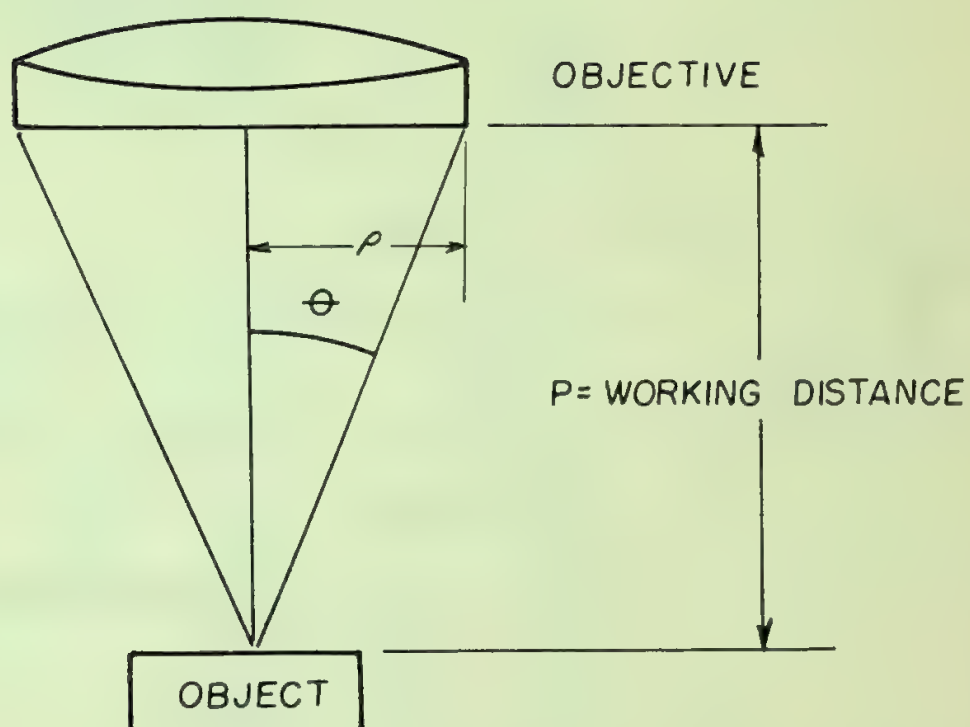


FIGURE 1
Parameters Relating to N.A. and
Working Distance

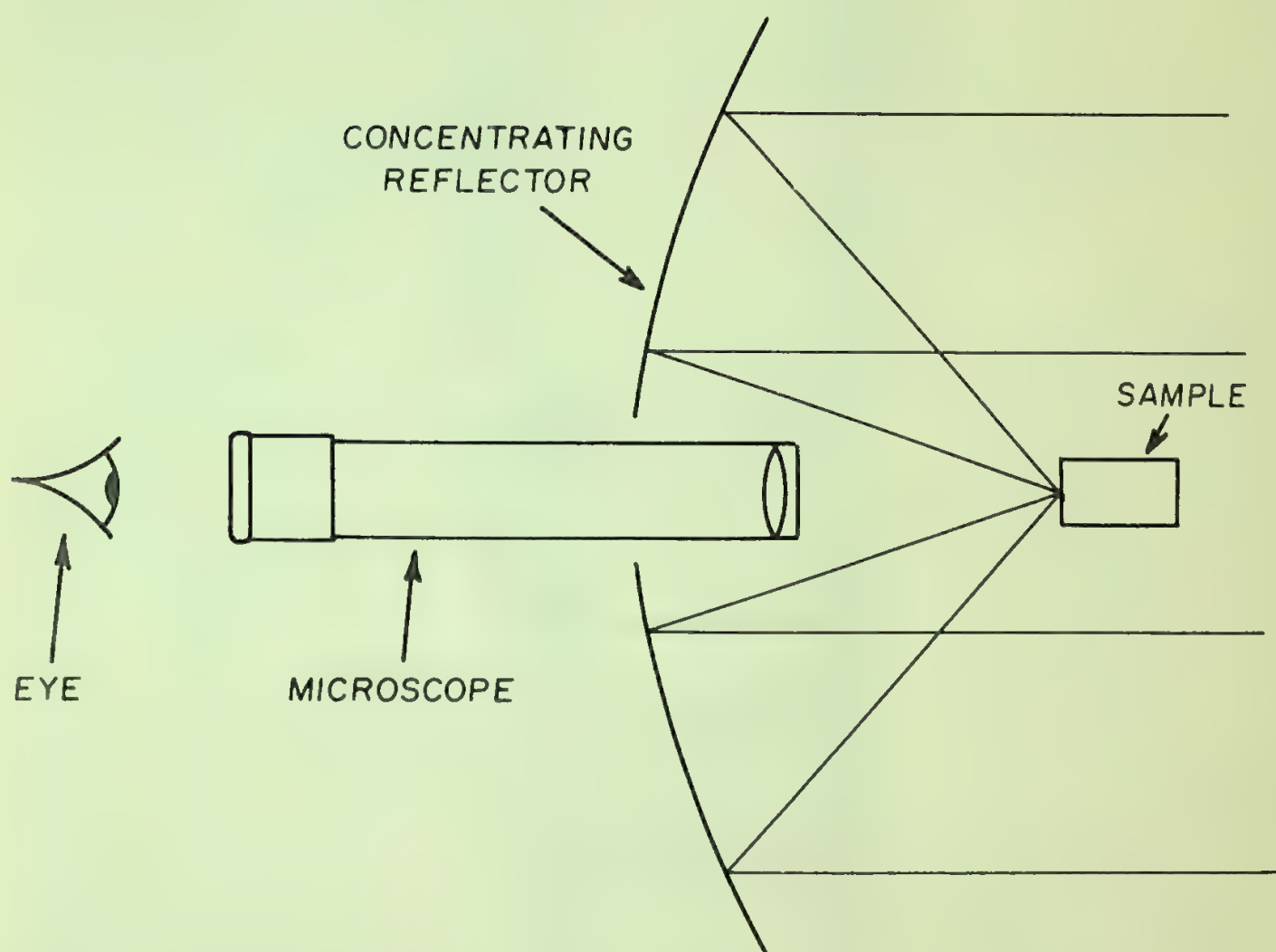


FIGURE 2
Generalized Microscope System
for Image-type Furnaces

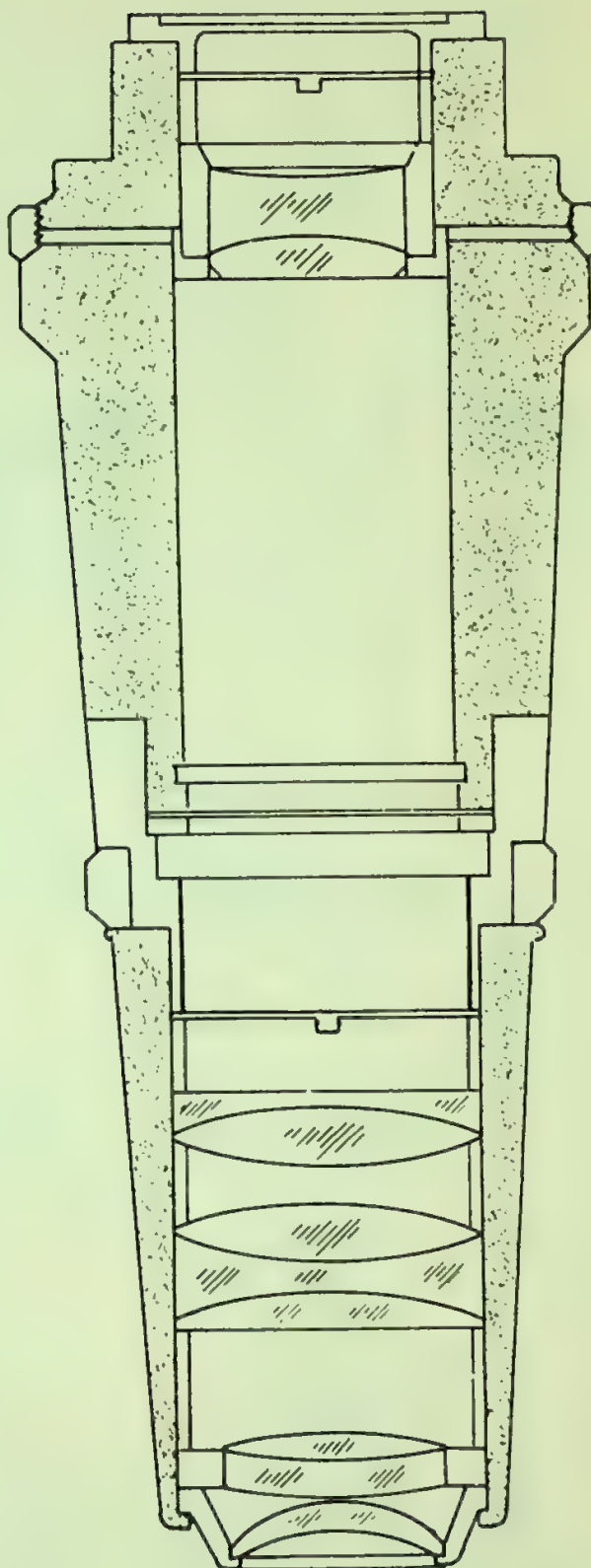
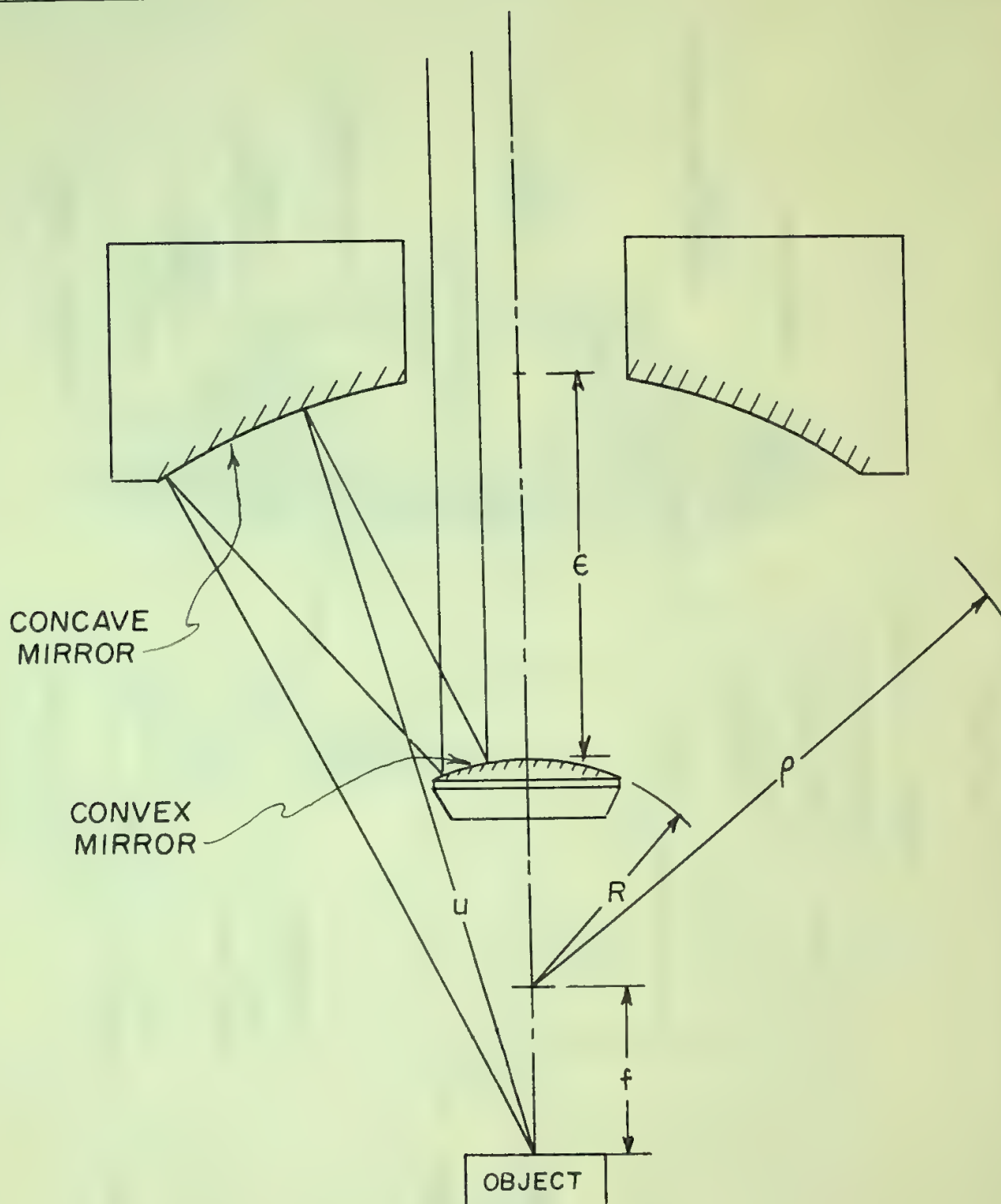


FIGURE 3

A Modified Conventional Objective,
Unitron FF40X



$$\epsilon/f = 2, \quad u/f = 2 + \sqrt{5}$$

$$\rho/f = \sqrt{5} + 1, \quad R/f = \sqrt{5} - 1$$

A Reflecting Objective
(Burch)

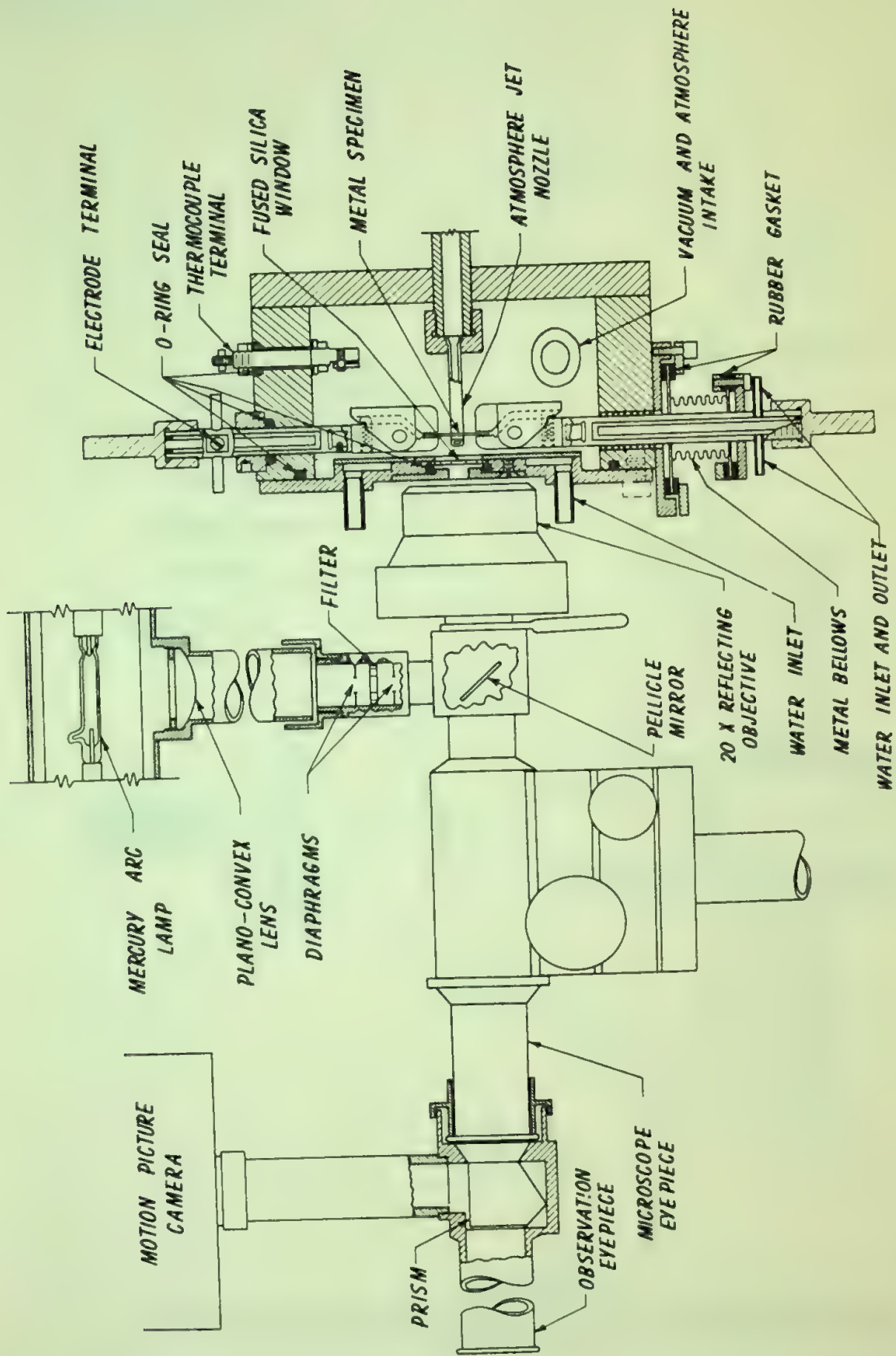


FIGURE 5
A Microscope System for Simultaneous Observation and Photography
(Steenstrup)

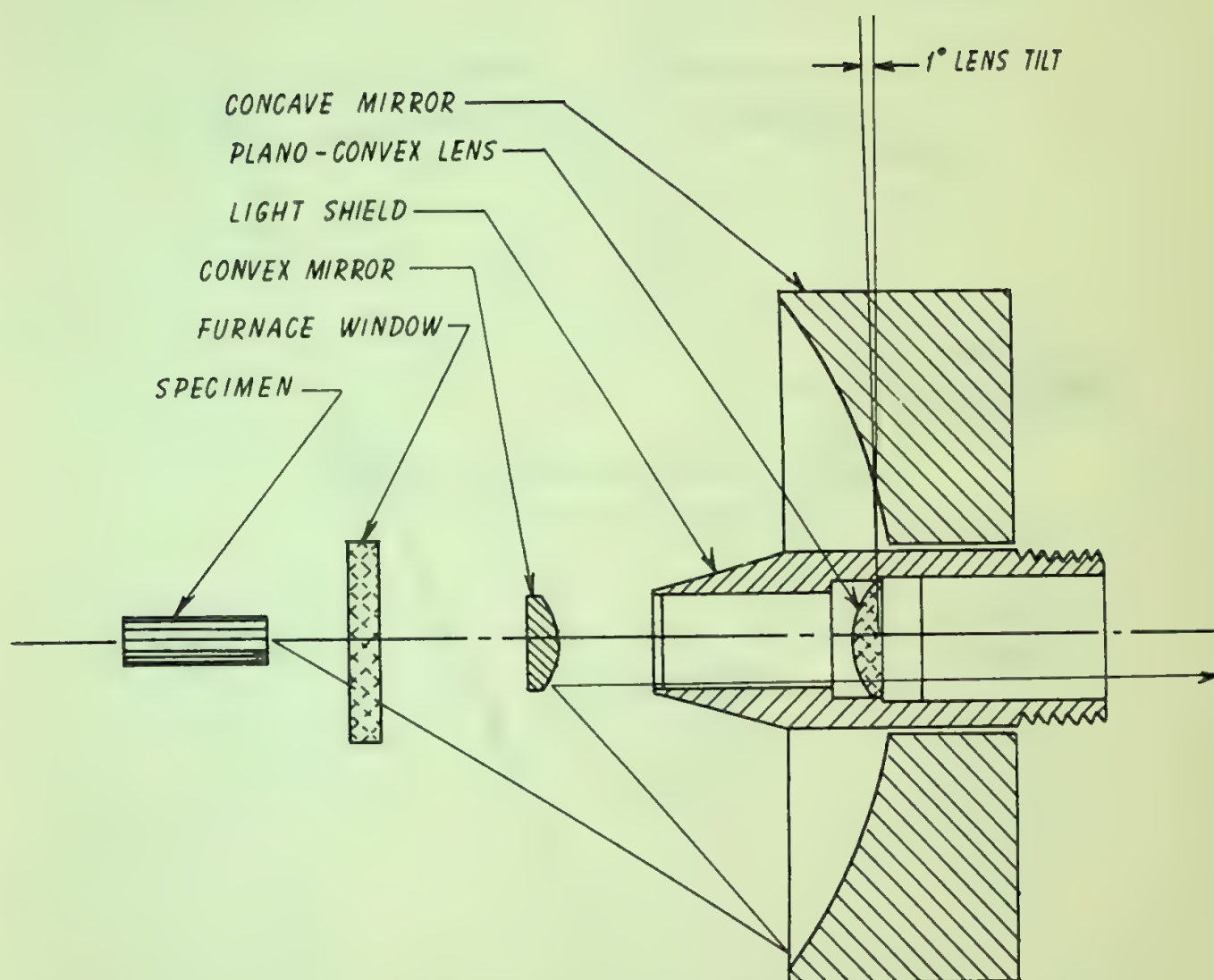


FIGURE 6

A Reflecting Objective with Zero or Low-power Lenses
(Jenkins, et al)

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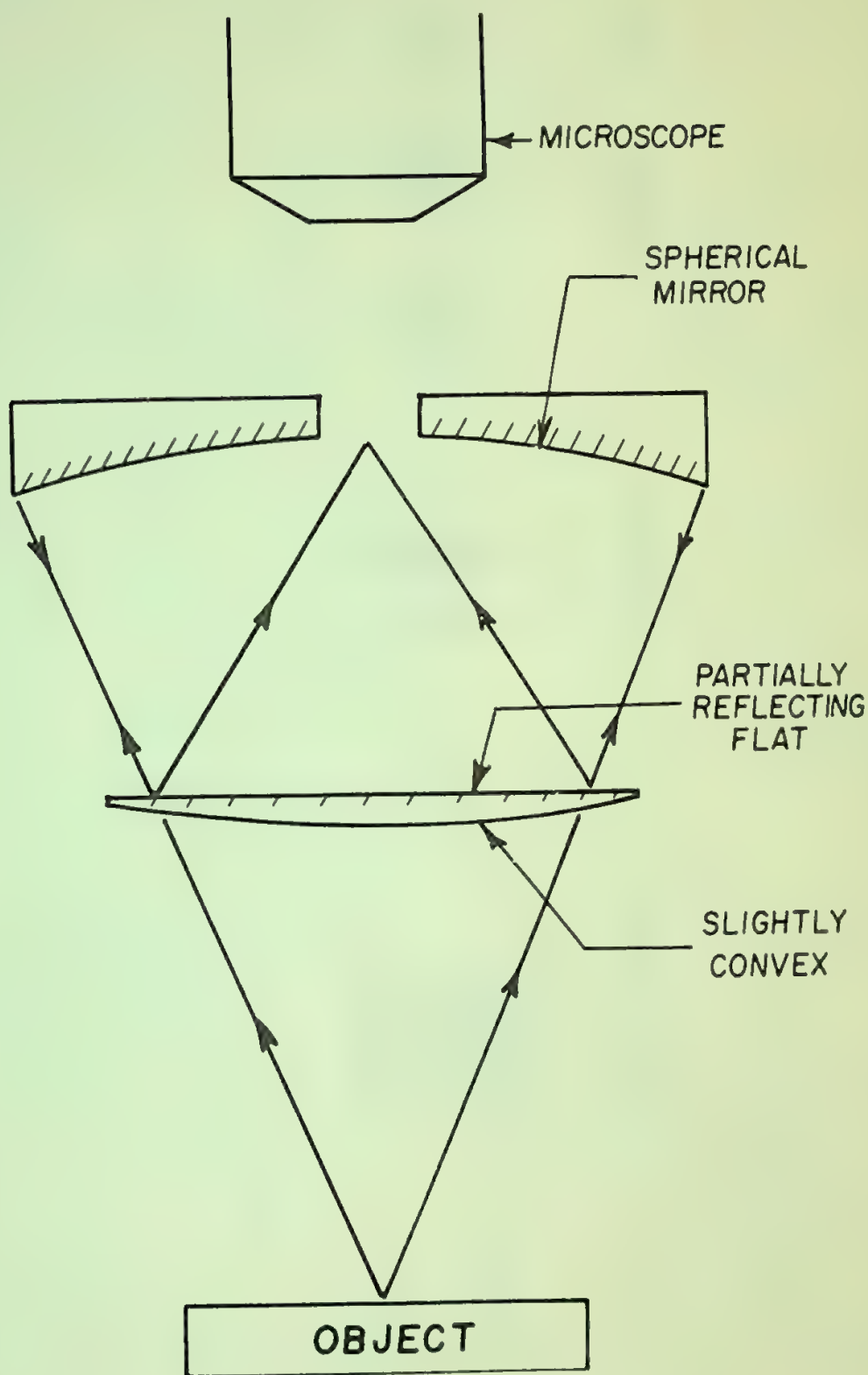


FIGURE 7

A Unit or Low-magnification Relay System

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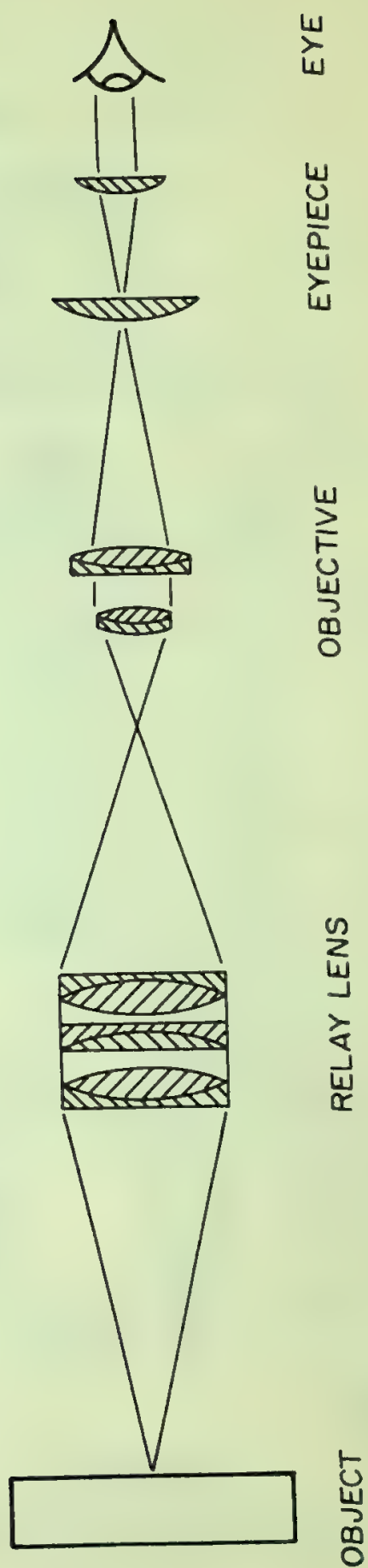


FIGURE 8
A Unit or Low-magnification Relay System
(Baumann)

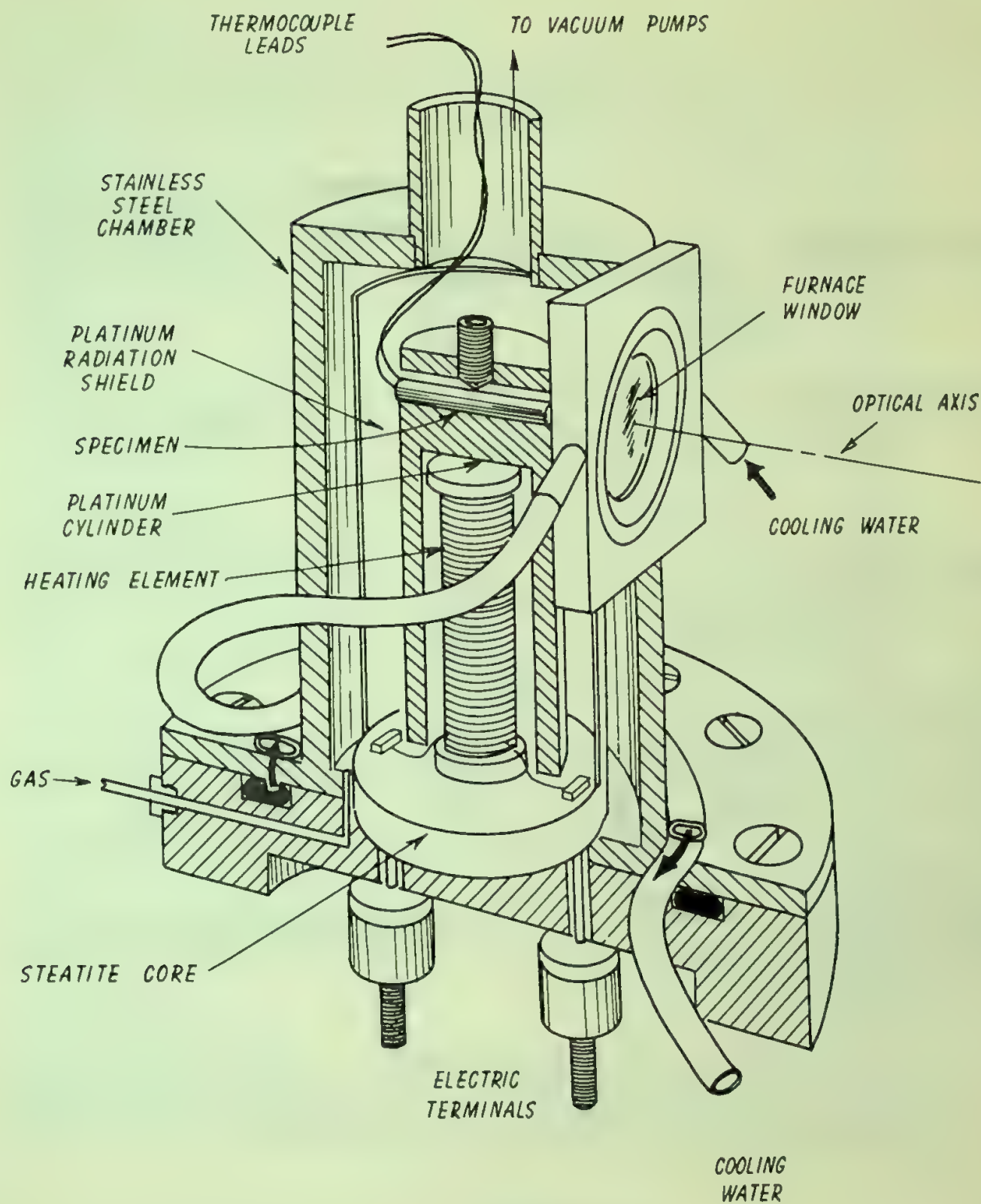
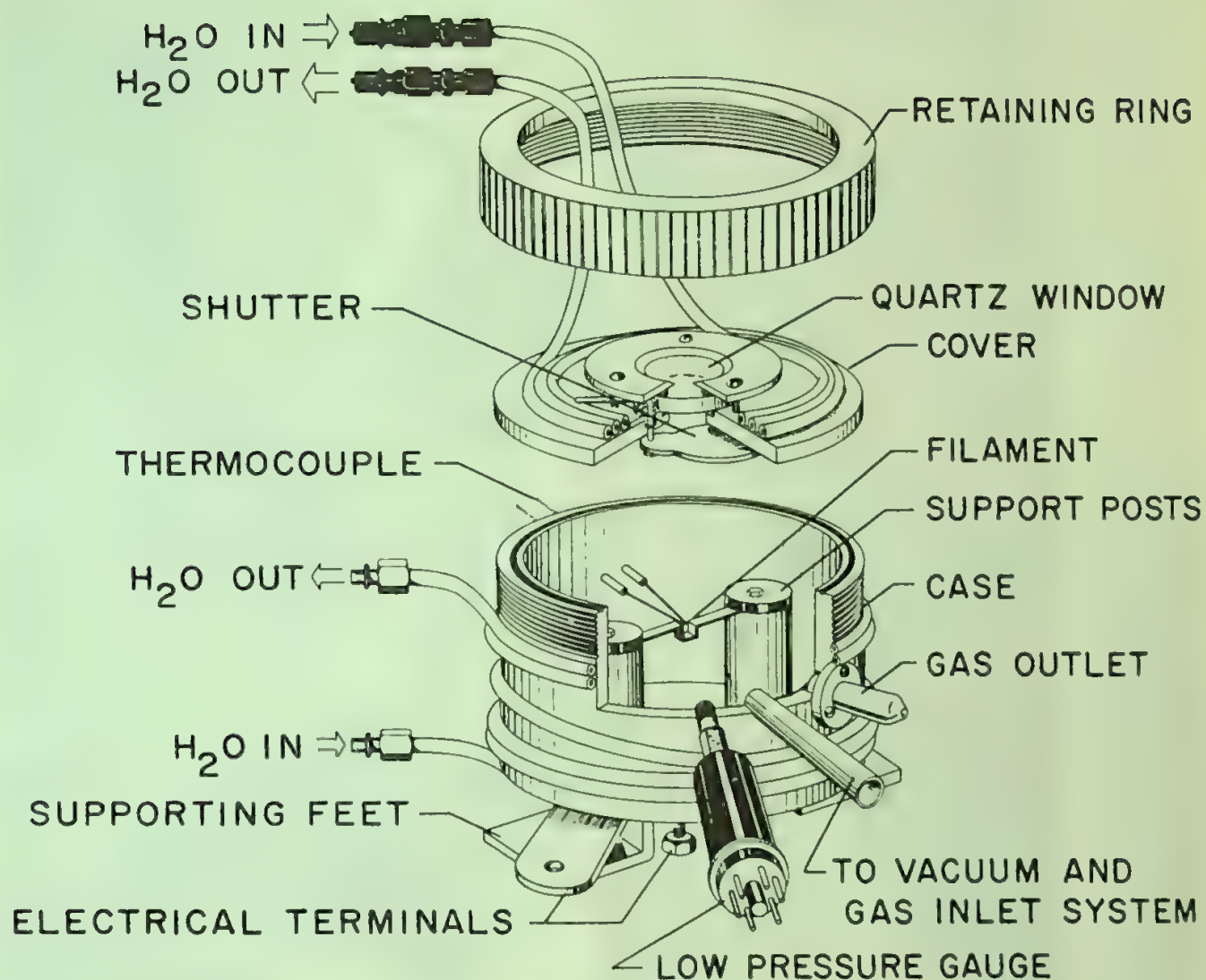


FIGURE 9
Self-resistance Heated Hot Stage
(Williams)



FILAMENT FURNACE

FIGURE 10
Tungsten Filament Furnace

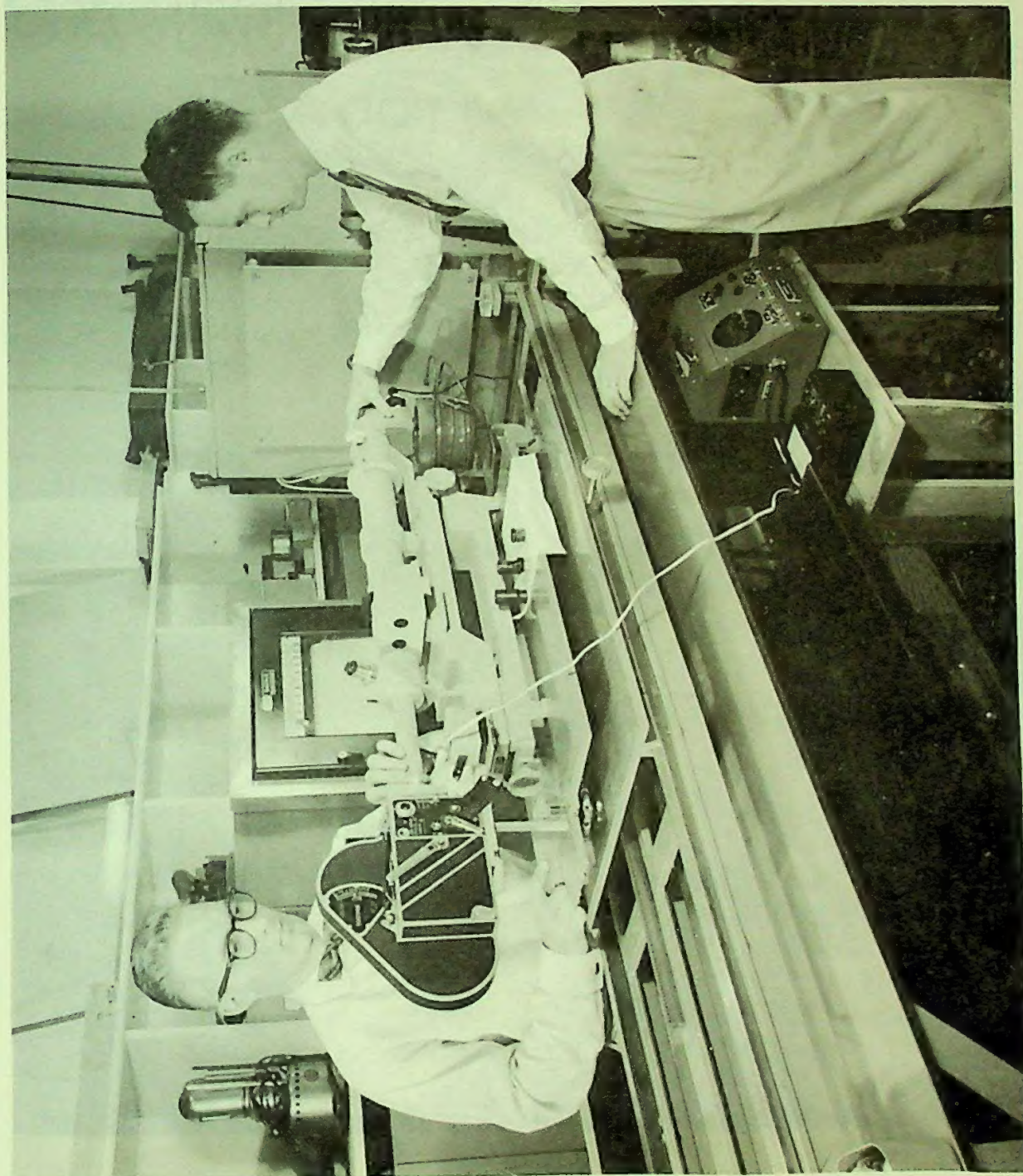


FIGURE 11
High Temperature Microscope, Tungsten Filament Furnace,
Carbon Arc Image Furnace, and Microscope

FIGURE 12

Photograph of Uranium Dioxide Crystals at 2850 °C
Taken Through American Optical Company Microscope



FIGURE 12

Photograph of Uranium Dioxide Crystals at 2650 C
Taken Through American Optical Company Microscope

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